A NEW DETERMINATION METHOD OF VERY LOW Fe CONTAMINATION BY UFS*

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ABSTRACT

We have produced very low Fe-dose (10^8 - 10^9 Fe/cm²) implanted Si samples. A new method of Ultraviolet Fluorescence Spectra (UFS) measurements has been used to determine the contents of Fe on the surfaces of Fe-samples. We find that the Fe-impurities have their own ultraviolet fluorescent peak wavelength at room temperature and their characteristic spectral peak intensities are proportional to the Fe-doses (Fe-concentrations) in Fe implanted Si samples. This method is very sensitive, efficient and nondestructive for testing the Fe contamination on silicon. Some SOI (Silicon on Insulator) wafers and VLSI chips were evaluated with the UFS method. The results indicate that the UFS method is able to measure the very low Fe-contamination. The limit is below (10^8 - 10^9) Fe/cm² or (10^{13} - 10^{14}) Fe/cm³ in Si samples.

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1. INTRODUCTION

The fabrication of today's most advanced integrated circuits is required to avoid some metal contamination. With shrinking device dimensions and thickness of insulating layers down to 100Å, the high content of metal impurities Fe, Ni etc. causes more failure and less reliability of semiconductor devices. The most traditional surface sensitive techniques, like Auger electron spectroscopy or X-ray photoelectron spectroscopy, are hampered by too high detection limit of about 10^{13} atom/cm² [1] [2]. With the total x-ray fluorescence (TXRF) method, absolute detection limits for transition metals are in the picogram range, corresponding to about 10^{11} atom/cm². It is difficult to determine the limit of 10^{10} Fe/cm² using the above analysis.

In this contribution, we report a new technique: Ultraviolet Fluorescence Spectra (UFS). We have found the impurities of Fe and Ni have their own ultraviolet fluorescent peak wavelength at room temperature. When the excitement ultraviolet wavelength $\lambda_{ex} = 350$ - 360 nm, there is an iron characteristic peak at the wavelength of $\lambda_{em} = 371, 383$ and 399 nm; when $\lambda_{ex} = 330$ - 350 nm, there is a nickel peak at $\lambda_{em} = 367$ nm.

The characteristic spectral peak intensities I_f are proportional to the content of the metal impurities C. They obey Stokes Law:

$$I_f = A\eta I_0(1 - e^{-KLC}) \quad (1)$$

In our case $KLC \ll 1$

$$I_f = A I_0 \eta KLC = A I_0 K * C \quad (2)$$

where η is the ultraviolet fluorescent efficiency of the metal; A is the illumination area on the

sample; I_0 is the excitement ultraviolet intensity; K and L are absorption coefficient and wavelength respectively. In this work, we measure the $\bar{A}$ (2.5mm^2), L (297nm), I_0 , and K^* using the UFS data from the standard wafers. Then we determine the Fe and Ni contents of SOI wafers by formula (1). We can obtain very high sensitivity up to $(10^{13}\text{-}10^{14})\text{ atom/cm}^3$ (about $10^8\text{-}10^9\text{ atom/cm}^2$).

2. EXPERIMENTAL RESULTS

2.1 Standard Fe-implanted wafers and UFS data

In order to get the Fe-standard sample, we use very careful cleaned wafers n-Si(100) 6-8 $\Omega\text{-cm}$, Fe implanted at 50 Kev with doses: 1.0×10^9 , 1.0×10^{10} , 1.0×10^{11} , 1.0×10^{12} and $1.0\times 10^{13}\text{ Fe/cm}^2$ respectively, then annealed at 140°C for 30 minutes. The corresponding concentration of Fe near the surface ($R_p=446\text{\AA}$) is 1.1×10^{14} , 1.1×10^{15} , 1.1×10^{16} , 1.1×10^{17} and $1.1\times 10^{18}\text{ Fe/cm}^3$, respectively.

Fig.1 shows the Fe-UFS results, the intensity I_f as the function of wavelength λ (nm), from the Fe-standard wafers by the ultraviolet fluorometer F-3010. It is clear that the intensity of the Fe-UFS peaks increases with the Fe content which obey the Stokes formula (1). We can obtain the ultraviolet fluorescent efficiency η and absorption coefficient K at three Fe-UFS peak wavelengths from Fig.1 and formula (1).

Fig.2 is the Fe-UFS intensity I_f at wavelength 371, 383 and 399 nm as function of the Fe-dose in the Fe standard samples.

Fig.1 Fe-UFS peak intensity I_f as function of wavelength ($\lambda=371, 383, 399\text{ nm}$) and Fe-dose ($10^9, 10^{10}, 10^{11}, 10^{12}, 10^{13}\text{ Fe/cm}^2$ and no-implantation) wafers from the Fe-standard wafer.

Fig.2 Fe-UFS peak intensity I_f at $\lambda=371, 383, 399\text{ nm}$ as function of Fe-standard dose ($10^9, 10^{10}, 10^{11}, 10^{12}$ and 10^{13} Fe/cm^2)

2.2 UFS data from SIMNI and SIMOX films

Four SOI-SIMNI (Separation by Implantation of Nitrogen) and eleven SOI-SIMOX (Separation by Implantation of Oxygen) wafers with different conditions were made in Erlangen, Surrey and Beijing Normal University. The UFS data were measured by the ultraviolet fluorometer F-3010 and RF-540 made in Japan. Using the data from Fig.1 and 2, we obtained the Fe contents on SIMNI and SIMOX films. Table 1 is Fe-UFS data and Fe contents from Germany SOI-SIMNI. The peak intensity at $\lambda=383$ nm from the surface of the sample is very low; the concentration of Fe is about $(2-3) \times 10^{14}$ Fe/cm³. The peak intensity at $\lambda=399$ nm from the backside of the samples is strong; the content of Fe is $(1-2) \times 10^{16}$ Fe/cm³. Table 2 is Fe-UFS data and contents from Germany SIMOX films and comparative wafer Japan n-Si(100). The concentration of Fe is $(2-4) \times 10^{14}$ Fe/cm³ (except SK20T) on the surface of the samples and $(1-3) \times 10^{16}$ Fe/cm³ on the backside surface. Table 3 is Fe-UFS data and contents from UK-SIMOX wafers and the comparative samples: US-Si, Japan Si. The Fe concentration near the surface is $(1.5-3.3) \times 10^{14}$ Fe/cm³; near the backside surface is $(7.2-8.3) \times 10^{16}$ Fe/cm³. Table 4 is Fe-UFS data and contents from BNU-SIMOX. The concentration of Fe is $(2.5-4) \times 10^{14}$ Fe/cm³ near the surface, $(1.8-2.4) \times 10^{16}$ Fe/cm³ near the backside surfaces, and 6.5×10^{14} Fe/cm³ near the polished-mirror-backside surface of No.3.

Table 1 Fe-UFS data from Germany SOI-SIMNI

No.	Surface Peak λ (nm)	Surface I_{λ} 2 ¹⁰	Surface 10^{14} Fe/cm ³	Back Peak λ (nm)	Back I_{λ} 2 ¹⁶	Back 10^{16} Fe/cm ³
SK2T	383	4.6-5	2.3	383	98	0.4
SK24T	383	4.6-5	2.3	399	200	1.0
SK21T	383	4.6-5	2.3	399	440	2.2
SK32T	383	6.0	3.0	399	426	2.1
US-Si	383	4.2	2.2			

Table 2 Fe-UFS data from Germany SOI-SIMOX

No.	Surface Peak λ (nm)	Surface I_{λ} 2 ¹⁰	Surface 10^{14} Fe/cm ³	Back Peak λ (nm)	Back I_{λ} 2 ¹⁶	Back 10^{16} Fe/cm ³
SK11T	383	8	4.0	399	272	1.4
SK12T	383	7	3.5	399	416	2.1
SK20T	383			399	392	3.0
SK16T	383	4	2.0	399	448	2.2

Table 3 Fe-UFS data from UK-SIMOX

No	Surface Peak λ (nm)	Surface I_{λ} 2 ¹⁰	Surface 10^{14} Fe/cm ³	Back Peak λ (nm)	Back I_{λ} 2 ¹⁶	Back 10^{16} Fe/cm ³
UK1	383	3	1.5	399	1664	8.3
UK-A	383	6.5	3.3	399	1632	8.2
UK-B	383	5	2.5	399	1446	7.2
US-Si	383	2.4	1.2			
Jap Si	383	7	3.5	399		0.01

Table 4 Fe-UFS data from BNU-SIMOX

No	Surface Peak λ (nm)	Surface I_{λ} 2 ¹⁰	Surface 10^{14} Fe/cm ³	Back Peak λ (nm)	Back I_{λ} 2 ¹⁶	Back 10^{16} Fe/cm ³
12-1	383	7	3.5	399	476	2.4
10-6H	383	5	2.5	399	216	1.1
11	383	6	3	399	368	1.8
3	383	8	4	387	13	0.065*
3-Si	383	6	3			

3. DISCUSSION

Concentration of Fe from the surfaces of all the SOI wafers is very low (about 10^{14} Fe/cm³). This indicates that the buried insulating layers (SiO_2 or Si_3N_4) have a strong function of absorption impurities Fe. The backside surfaces of all the SOI wafers are weakly polluted. The content of Fe is about 10^{16} atom/cm³. This means that the rough surface on backside of SOI wafers is easily polluted during the SOI performance. The polished backside surface (as a mirror surface) of the SOI wafer has very low content of Fe about 10^{14} - 10^{15} Fe/cm³. The surface of VLSI chips has been examined by UFS method. The content of Fe depends of their fabrication environments. Some VLSI chips have very high contamination of Fe up to 10^{18} Fe/cm³, which causes the failure of the devices. Ultraviolet Fluorescence Spectra (UFS) measurements is a very sensitive method of determining the contamination of Fe on the surface of semiconductor wafers, with limit down to 10^{13} - 10^{14} Fe/cm³ (about 10^9 Fe/cm²).

Reference

- [1] V. Penka and W. Hub, Spectrochimica Acta, Vol.448, No.5, 483, 1989
- [2] W. Bergholz, Silicon Clean Processing, Oxford, Sep. 1991.